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FRACTURE CHARACTERISTICS OF ALUMINUM ALLOYS WETTED WITH LIQUID ZINC AMALGAM

by

B. J. ROGUS

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FRACTURE CHARACTERISTICS OF ALUMINUM ALLOYS WETTED WITH LIQUID ZINC AMALGAM

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ABSTRACT

Fracture strength determinations were made on sheet specimens of aluminum alloys 1100, 2024, 5456, 6061, and 7075 in different conditions of temper after wetting with a liquid 2% zinc amalgam. Tests at ambient temperature disclosed that the wetted fracture strength of the alloys decreased with increasing time of wetted contact with the amalgam before stressing. This time dependence was most pronounced for alloys in the precipitation hardened condition in comparison to that for the same alloys in the annealed state or others in strain hardened tempers.

Once wetting had been effected, there was a continued physical dissolution attack of the aluminum alloys by the amalgam. This attack, however, was not directly related to the noted time dependence of the wetted fracture strengths.

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INTRODUCTION

The embrittlement of aluminum alloys by pure mercury and by amalgams has been the subject of considerable study. Of interest is the fact that the degree of embrittlement can vary greatly from alloy to alloy. Efforts made to clarify those factors which influence the severity of the process for specific couples have led to a better understanding of the entire phenomenon.

In a summary of many investigations on this subject presented in a book by Rostoker, McCaughey, and Markus(1)*, degree of embrittlement induced in aluminum was shown to be related principally to the strength level of the alloy. High strength level alloys were seen to be most severely affected. Later work by Rostoker(2) showed that the sequence of prior thermal and mechanical processes used to obtain the strength level of the alloy was important. Different sequences of cold working and thermal treatment were used on specimens of alloy 2024 to obtain groupings of comparable strength levels. However, for these specimens the observed degree of embrittlement was seen to vary greatly depending on the sequence of prior operations, even though the strength levels for all groupings were approximately equal.

Previous work on the effect of time on the embrittlement process has been limited mainly to the delayed fracture aspect of the process. In this regard, a separate study was performed at Frankford Arsenal(3) on the delayed fracture of aluminum alloys in the presence of a liquid 2% zinc amalgam.

For this earlier program, tests were made on specimens which had been stressed to selected levels before wetting with the amalgam. Delayed fracture times were then recorded to failure. Results of this previous study showed that delayed fracture of precipitation hardened aluminum alloys 2024 and 7075 could be effected at room temperature. In addition it was shown that no degradation of mechanical properties was evidenced for the wetted specimens up to the instant of failure.

A significant aspect of behavior was the fact that brittle fracture of the aluminum was accomplished after the passage of finite time intervals. Therefore, to more fully understand the phenomenon, it was deemed necessary to examine the role of time in embrittlement more closely.

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*See References.

The present investigation was undertaken to determine to what extent the embrittlement process is time dependent. Noted relationships between strength level of the alloy, history of thermal-mechanical treatment, and the observed embrittlement behavior may be affected by the factor of time. Clarification of such information could prove most useful in establishing a satisfactory comprehensive model for liquid metal embrittlement phenomena.

MATERIALS

Commercial quality sheet material of different aluminum alloys were investigated. A variety of alloys were used in order to show whether chemistry or processing treatments were determining factors in time dependence of fracture strength. Selection of materials was made to include unalloyed 1100 aluminum; precipitation hardened grades 2024 T-3, 6061 T-6, 7075 T-6; strain hardened 5456 H-24 and H-343 tempers; as well as age hardenable grades 2024 and 7075 in the annealed "0" condition. In addition, alclad 2024 T-3 alloy was studied. Thickness of the different sheet alloys ranged from 0.040 to 0.092 in.

The material was tested as received or after heat treatment as described. Conventional tensile properties of the alloys in the unwetted condition are listed in Table I.

A liquid 2% zinc amalgam was used to embrittle the alloys. As explained in the earlier report[3], selection of an amalgam rather than pure mercury was based on its superior wetting characteristics. It is believed that the presence of zinc is not a controlling factor in the occurrence of time dependence behavior. Exploratory tests, employing chemical wetting, showed that similar effects were encountered with mercury as with the zinc amalgam.

Table I. AVERAGE TENSILE PROPERTIES OF ALUMINUM SHEET MATERIAL IN THE UNWETTED CONDITION

Alloy and Temper	Yield Strength (psi) (0.2% Offset)	Tensile Strength (psi)	Elongation (% in 2")
1100-0	5,050	13,700	36.6
2024-0	11,000	33,000	22.5
2024 T-3	51,800	71,400	19.5
2024 T-3 Alclad	53,100	71,100	17.7
5456 H-24	44,100	57,300	11.3
5456 H-343	48,600	59,800	7.4
6061 T-6	41,200	44,700	11.2
7075-0	15,000	34,000	18.5
7075 T-6	73,700	83,700	9.5

EXPERIMENTAL PROCEDURE

Tensile tests were conducted at room temperature using flat specimens machined from the different alloy sheet materials. Test specimens were machined to 2-inch gage length by 1/2-inch gage width dimensions.

A table tensile machine was used which permitted stressing the specimens while held horizontally. In this manner the amalgam lying on the specimen gage section would not run off during test. The test machine was screw activated and was equipped with friction grips. Special sliding cross arm supports were designed and installed to support the jaws so that their weight would not impart any bending stresses to the specimens. Preliminary checks made on specimens with SR-4 strain gages on either side of the gage section confirmed that there was no bending of the samples in test and that the loading was essentially uniaxial tension.

A SR-4 strain gage load cell was installed in the system and calibrated to measure applied loads. Accuracy of the load measuring arrangement was verified to be ± 1 percent of the indicated value. A photograph of the test arrangement is shown in figure 1.

Test procedure was as follows. While in the unstressed condition, test samples were wetted in the center of the gage section with the liquid 2% zinc amalgam. The specimens were allowed to remain in the unstressed state at room temperature for predetermined periods of time. After selected time intervals the pieces were loaded to failure and fracture strength values recorded.

By wetting is meant the removal of penetration of intervening oxide or other surface layers present between the aluminum and the amalgam and the subsequent formation of a true interface between the solid aluminum and the liquid wetting metal.

Wetting was accomplished both mechanically and chemically. For mechanical wetting, an ultrasonic vibrating probe was used, the tip of which vibrated at 28 KC. Five drops of amalgam were placed on the specimen gage section and the probe inserted into the pool. Upon activation, the vibrating tip effectively broke down the thin oxide layer on the aluminum and allowed the amalgam to wet the specimens. Effective wetting could be discerned both by the emission of a distinct hissing sound from the amalgam pool and by visual observation of the manner in which the amalgam spread over the aluminum surface. The probe was passed through the amalgam over the gage section for 30 seconds. Care was exercised not to touch the specimen surface with the probe tip, for experiments showed that erratic wetted fracture strength values are thereby obtained. In addition, the amalgam was kept away from the sides of the specimens to avoid possible misleading edge effects.

For chemical wetting a combination of 10% sodium hydroxide solution and amalgam was used. Five drops of NaOH solution were placed on the center of the specimen gage section. After 30 seconds, during which time the NaOH dissolved the oxide film on the aluminum, five

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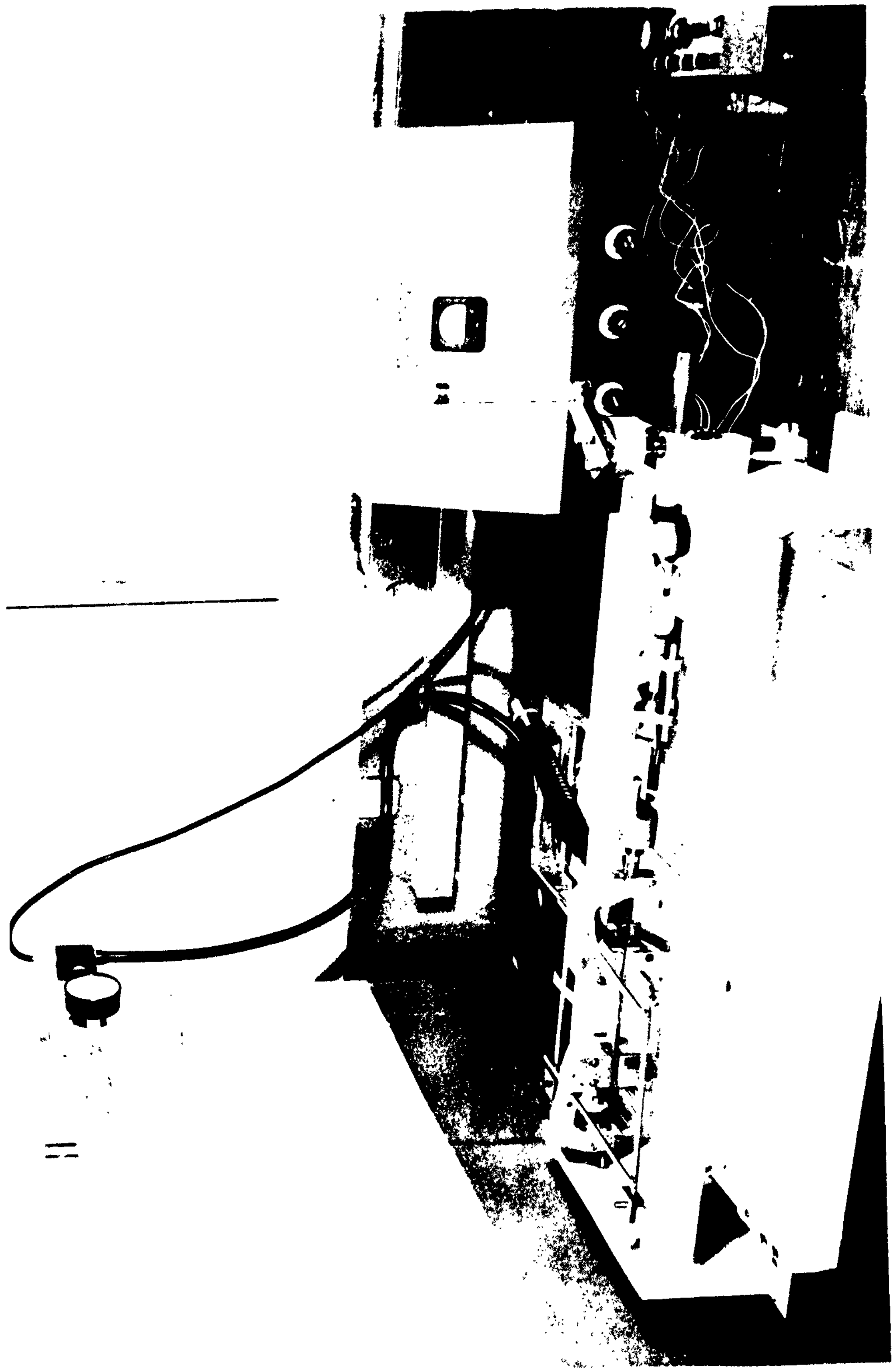


drops of amalgam were added into the center of the NaOH pool. The oxide film, having been removed, the amalgam was able to make contact with the aluminum base metal and thereby effect wetting.

Preliminary tests had shown that the presence of the NaOH solution alone, without any amalgam, had no measureable effect on the aluminum fracture strength values within the time intervals studied. Moreover, the use of chemical wetting was limited to the study of alloy 2024 T-3. This was done only to confirm whether behavior encountered with mechanical wetting could also be effected through chemical methods.

RESULTS AND DISCUSSION

Fracture Strength as a Function of Time Wetted Prior to Stress

For each alloy, wetted fracture strength values were determined by loading specimens to failure immediately after the completion of wetting. Obtained results were in line with those reported by other investigators. Sub-yield fracture was recorded for alloys in the precipitation hardened condition while failure of annealed or strain hardened material occurred at stress levels above the yield strength.

Other test specimens were wetted in identical manner as those discussed above. However, these were held in the unstressed condition for periods of time before loading. Wetted fracture strength values obtained on the alloys as a function of time are given in Table II.

Precipitation Hardened Alloys

Tests on samples from alloy 2024, 6061, and 7075 in the precipitation hardened condition showed that decreases in wetted fracture strength occurred with increased holding times prior to stressing. The decline in breaking stress values with time was considerable. For example, after holding times of 60 minutes, fracture could be effected on 2024 T-3 specimens at stress levels as low as 12,000 psi. For the same material, wetted fracture strength values of 47,000 psi were obtained immediately after wetting.

Table II. AVERAGE WETTED FRACTURE STRENGTH VALUES, PSI, OBTAINED ON ALUMINUM SHEET SPECIMENS. SPECIMENS WERE HELD UNSTRESSED AFTER WETTING MECHANICALLY WITH A 2% ZINC AMALGAM.

	Time Held Wetted Before Stressing (Min.)						
Alloy	Immediate	5	15	30	60	240	1080
1100-0	12,500					12,000	
2024-0	31,500				30,000		28,000
2024 T-3	47,000	31,000	12,400	12,300	11,700		7,400
2024 T-3 Alclad	50,000		50,400	51,050			10,400
5456 H-24	51,000	49,750		49,000			40,300
5456 H-343	40,900	35,750		39,500			33,600
6061 T-6	41,850	27,400		25,700			15,700
7075-0	28,100				28,350		26,300
7075 T-6	31,300			24,200	17,000		10,600

Wetted fracture strength test values obtained on the precipitation hardened alloys are plotted in figures 2 and 4 while bar graph representations are shown in figures 3 and 5.

Although the induced embrittlement was effectively made more severe as the material was held wetted for periods of time, no progressive degradation of the materials properties themselves was evidenced. This was shown by tensile tests made on specimens upon removal of the amalgam. Specimens of alloy 2024 T-3 which had been held wetted for six days (wetted fracture strength values after this exposure were less than 10,000 psi) were dewetted by washing with water and subjected to conventional tests. Although some physical dissolution had taken place during exposure, obtained tensile strength values, when compensated for the noted reduced thickness, were comparable to those obtained on unwetted material.

Experiments were conducted to show whether the described time dependence phenomenon could be the result of surface effects caused by mill handling conditions. Uneven stress distribution patterns could possibly result which might influence embrittlement behavior. To investigate this aspect, specimens from alloys 2024 and 7075 were reheat treated and then aged. For these reheat treated specimens similar decreases in stress required for fracture were recorded for increased holding times.

Of interest was the consideration of the ultimate extent of time dependence of wetted fracture strength. In this regard, specimens from 2024 T-3 material were allowed to remain six days while in the wetted condition before loading to failure. Values obtained on these specimens were approximately equal to those recorded after holding times of 18 hours. This observation indicated a possible lower limit for the effect.

Annealed and Strain Hardened Alloys

Tests on annealed 1100-0, 2024-0, and 7075-0 material showed but a slight decrease in wetted fracture strength values with time. In no instance did the obtained fracture strength values drop below the alloys annealed yield strength. It was noted that for holding times greater than approximately five minutes, alloy 2024 in the annealed "0" condition showed greater fracture strength values than did the same alloy in the precipitation hardened T-3 condition.

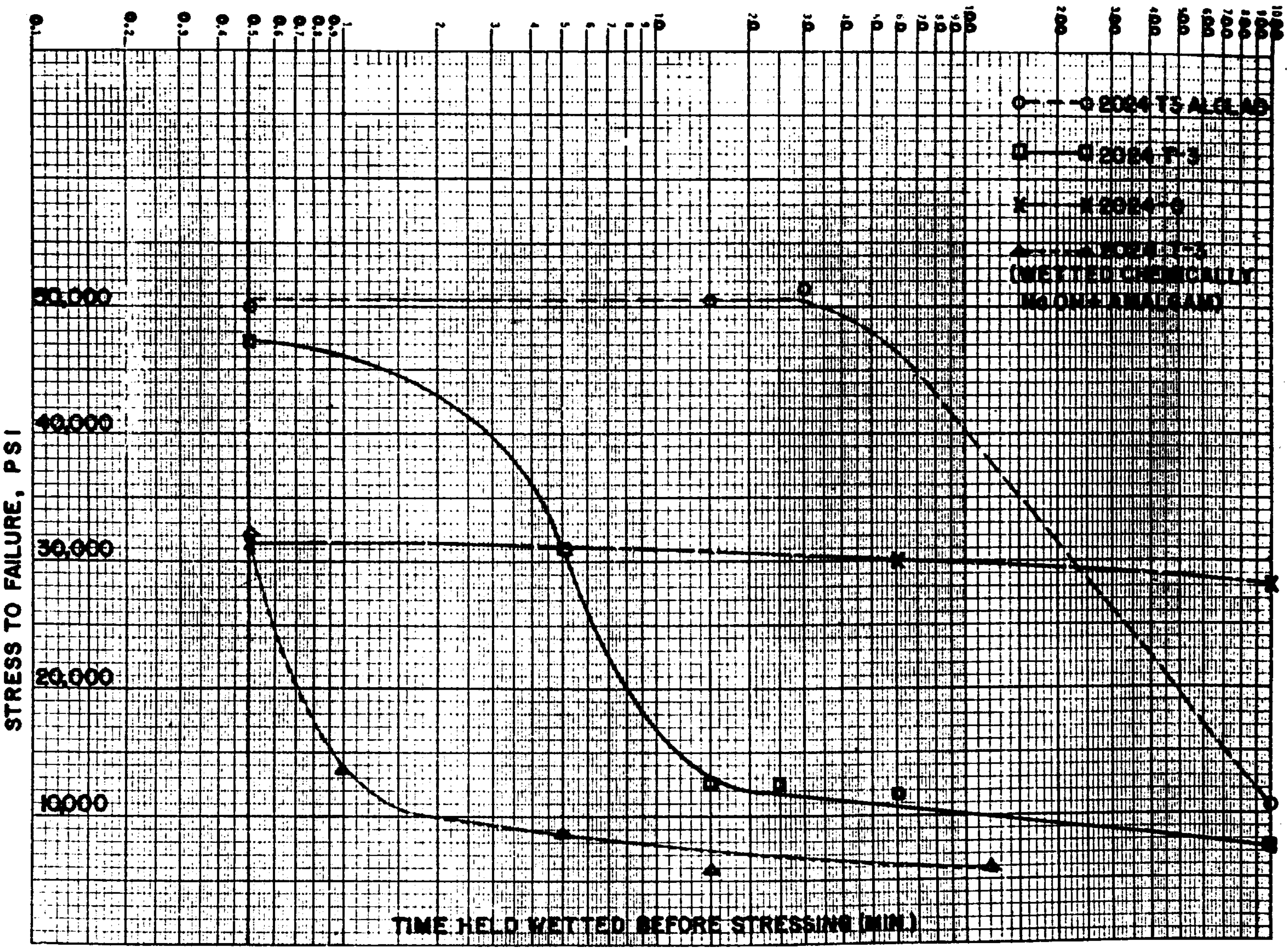


Figure 2. Wetted fracture strength of aluminum alloys as a function of time held wetted unstressed before loading. Specimens wetted mechanically with 2% Zn - Hg amalgam except as noted.

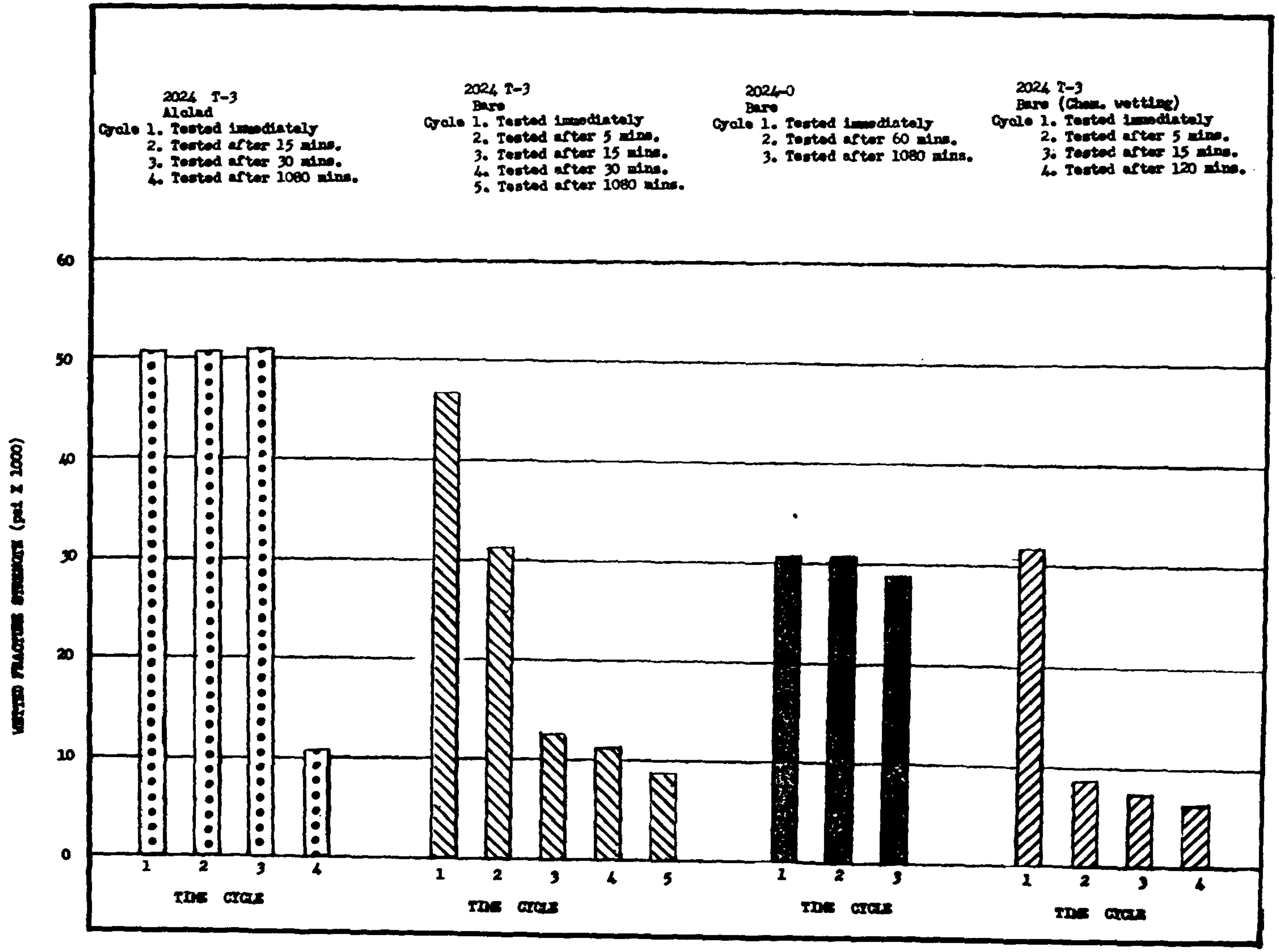


Figure 3. Change in wetted fracture strength as function of increasing time held wetted before stressing. Specimens wetted mechanically with 2% Zn - Hg amalgam except as noted.

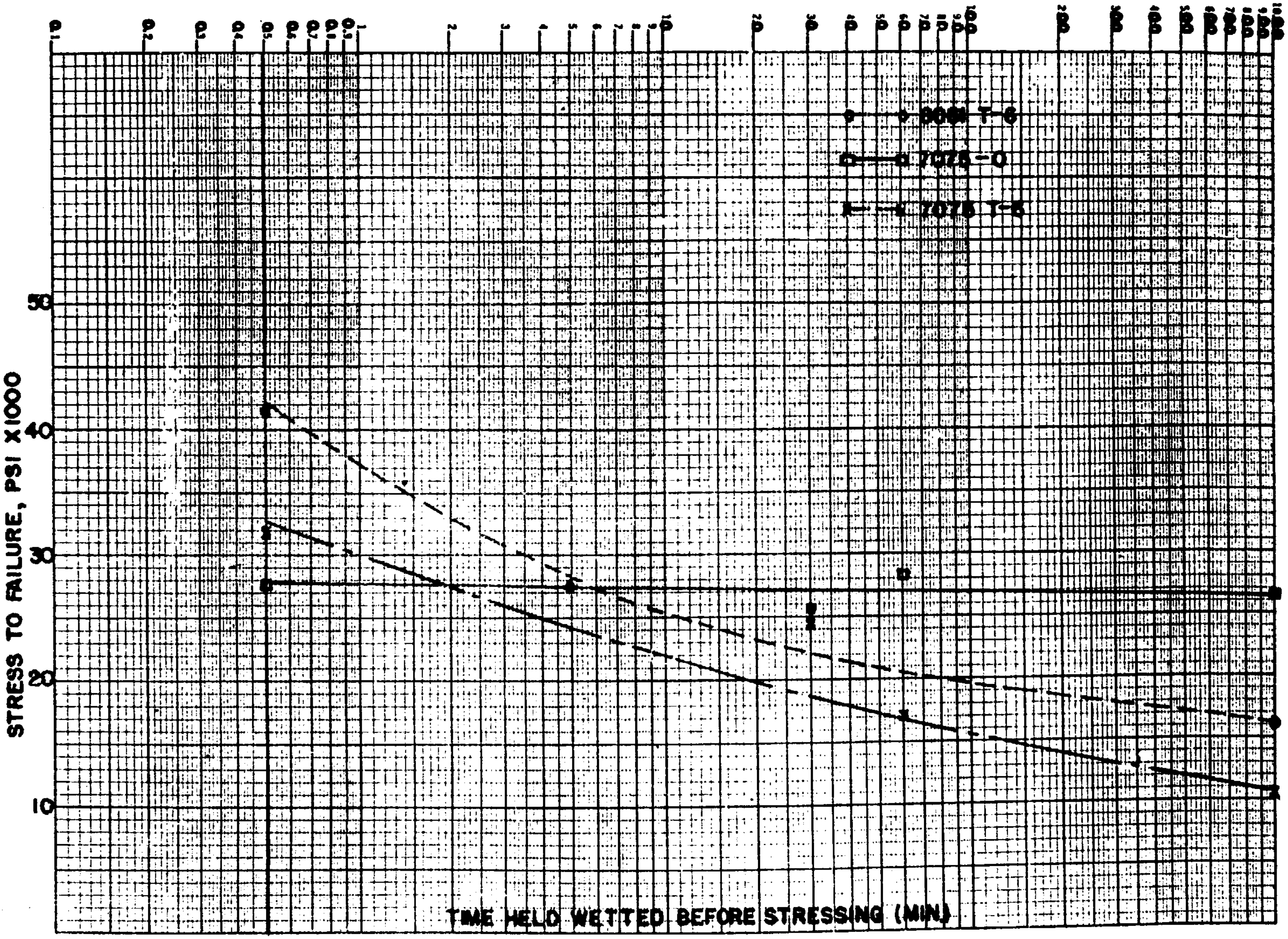


Figure 4. Wetted fracture strength of aluminum alloys as a function of time held wetted, unstressed, before loading. Specimens wetted mechanically with 2% Zn - Hg amalgam.

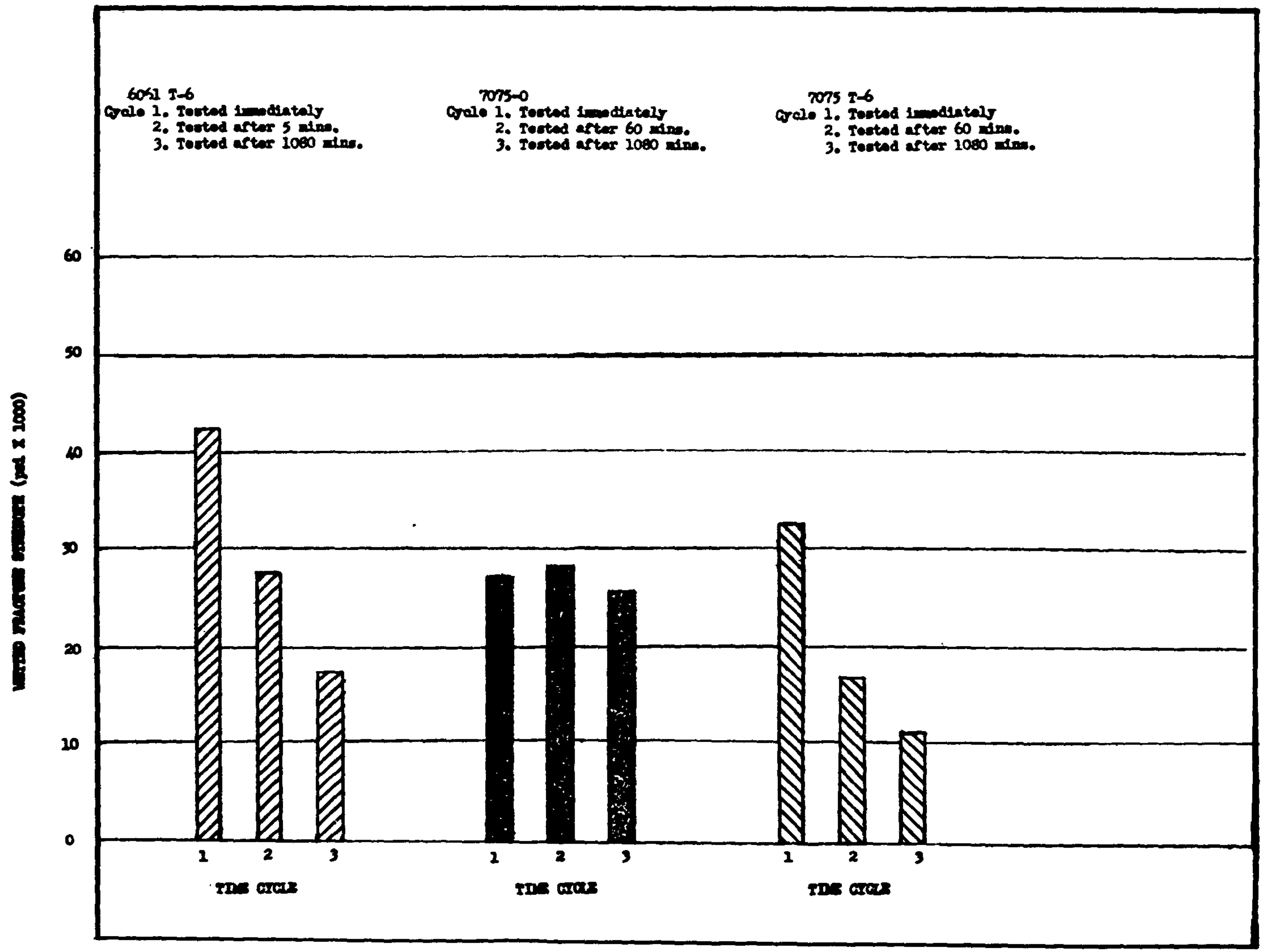


Figure 5. Change in wetted fracture strength as function of increasing time held wetted before stressing. Specimens wetted mechanically with 2% Zn - Hg amalgam.

Decreases in wetted fracture values for strain hardened 5456 alloy were much less than that exhibited by the precipitation hardened material, but more pronounced than that for annealed aluminum. Data for the annealed and strain hardened alloys are presented graphically in figures 2 through 7.

Clad Material

As mentioned previously, unalloyed 1100-0 aluminum did not show any significant time dependence of wetted fracture strength. Therefore, to investigate the possible effect of a surface layer of such material, tests were conducted on Alclad 2024 T-3 material. Obtained wetted fracture strength values were compared to those of bare stock. The clad material exhibited somewhat different behavior than that of the bare sheet. For times up to 30 minutes during which the material was held unstressed, the subsequent wetted fracture strength values upon loading remained basically unchanged. After 30 minutes the clad material showed an average wetted fracture strength of 50, 000 psi whereas specimens from bare sheet, similarly held, broke at an average stress of 12, 300 psi. Wetted fracture strengths obtained immediately after wetting were 50, 000 psi and 47, 000 psi for the clad and bare material respectively. For time increments greater than 30 minutes there was a decrease in stress required to fracture the clad material so that after 1080 minutes the values obtained on the clad material were basically similar to those of the bare, approximately 10, 000 psi.

Values for the clad and bare 2024 T-3 material are shown plotted in figure 2. Bar graph representations are shown in figure 3.

Chemical Wetting

Additional tests were made on bare alloy 2024 T-3 using chemical wetting. This was done to confirm that behavior encountered with mechanical wetting could also be accomplished with chemical methods. Results for chemical wetting are plotted in figure 2 and shown graphically in figure 3.

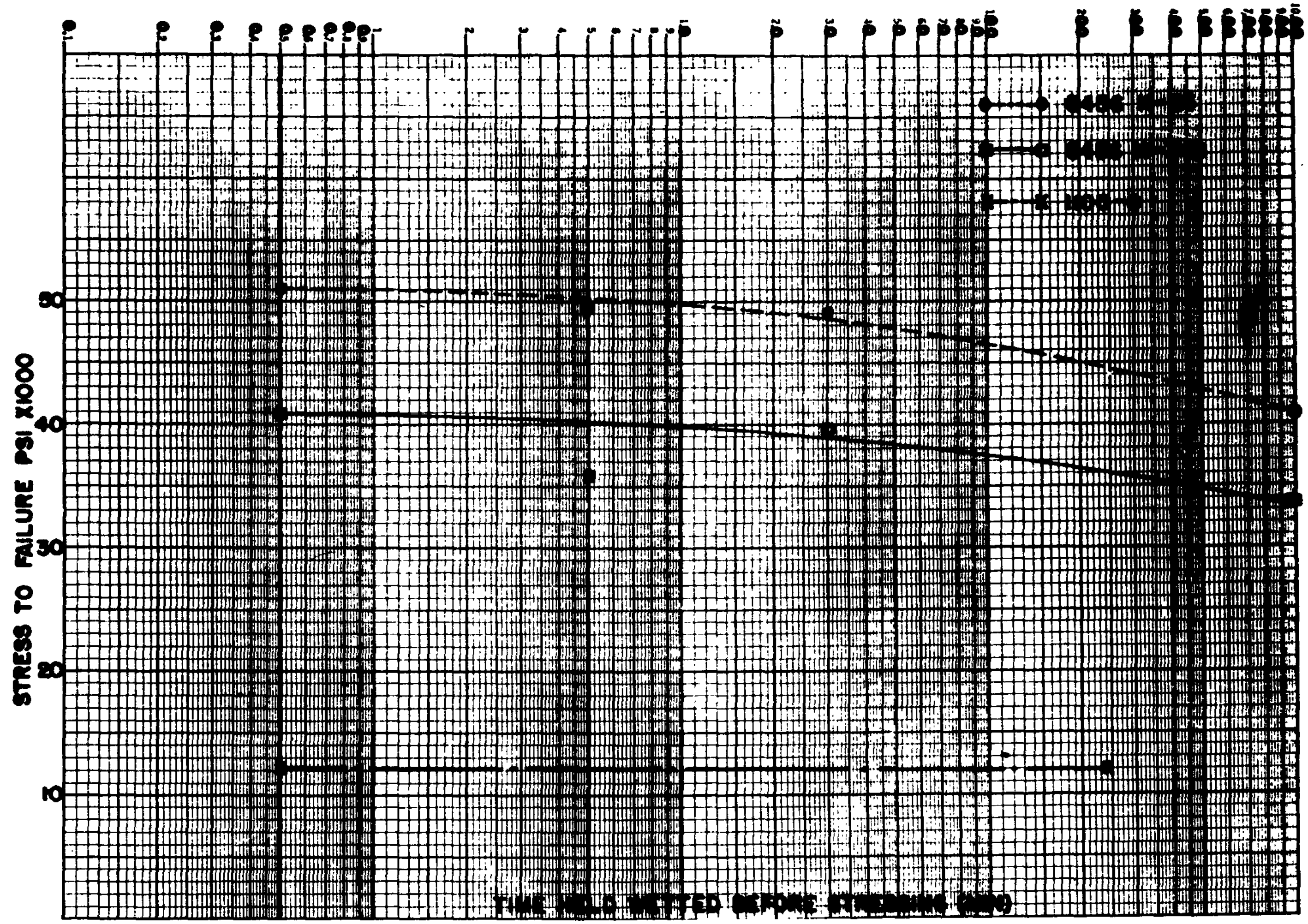


Figure 6. Wetted fracture strength of aluminum alloys as a function of time held wetted, unstressed, before loading. Specimens wetted mechanically with 2% Zn - Hg amalgam.

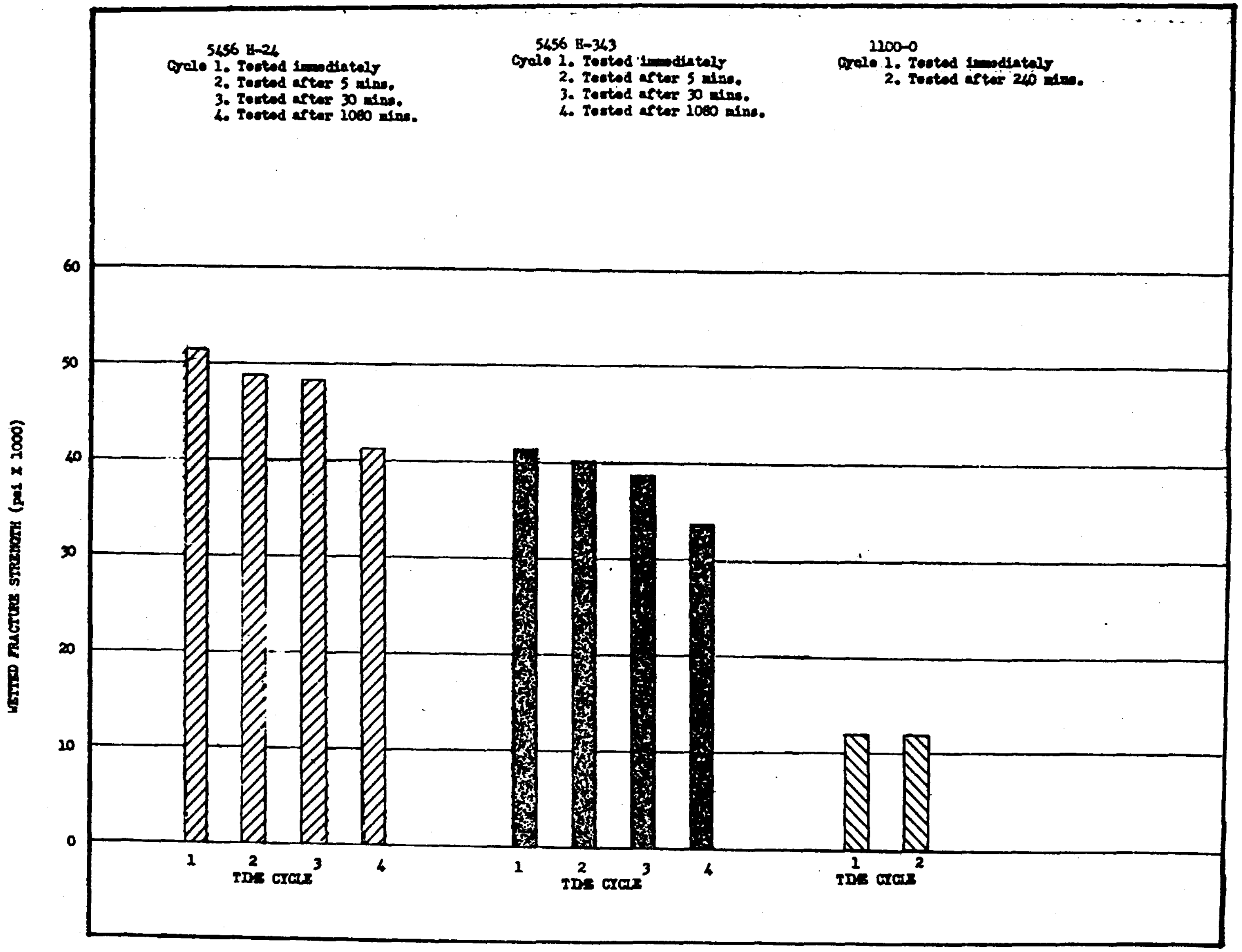


Figure 7. Change in wetted fracture strength as function of increasing time held wetted before stressing. Specimens wetted mechanically with 2% Zn - Hg amalgam.

Metallographic Studies

The time dependence of wetted fracture strength values for the aluminum alloys was considered significant. Test values indicate that the fracture strength was effectively reduced as the material was held wetted for periods of time. However, the fact that time dependence was much greater for age hardened alloys in comparison to that for annealed or strain hardened material suggested additional complexities. Factors related to strength level or thermal-mechanical history of the alloys possibly are involved.

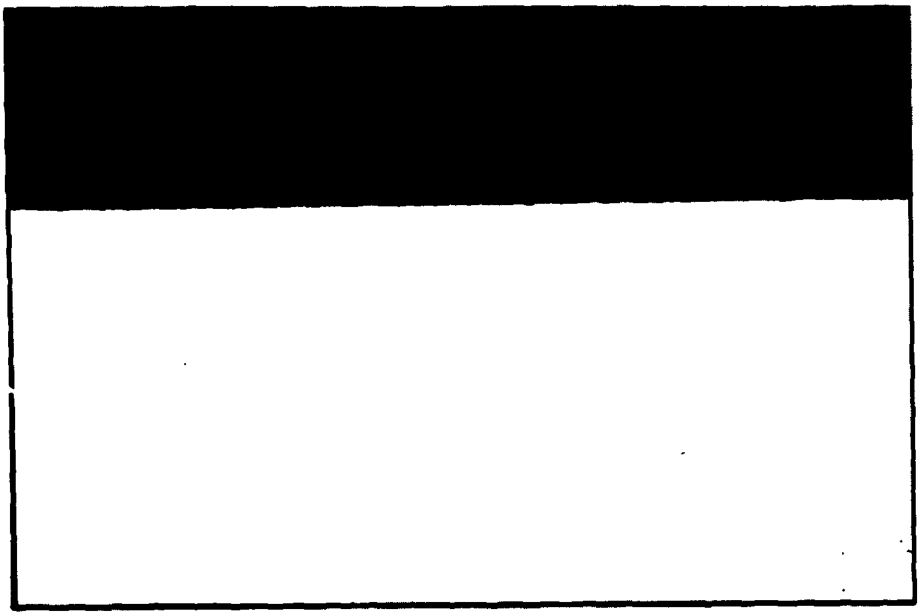
To investigate whether the time dependence process could be clarified by microscopic means, specimens from the various alloys were examined metallographically. A series of photomicrographs was prepared to study the effect of the amalgam on the aluminum alloy surfaces as a function of time.

Precipitation Hardened Alloys

Specimens from alloy 2024 T-3 were wetted with amalgam using the ultrasonic probe and were then dewetted at increasing intervals and subjected to microscopic examination to detect extent of amalgam penetration. As shown in the photomicrographs (see figures 8 through 14) there was a physical, dissolution of the aluminum surface by the amalgam which progressed with time.

This attack appeared to take the form of penetration of amalgam and lifting out of whole grains. Generally, penetration was not deep. The rate of dissolution was not very rapid; after 72 hours wetted exposure the measured depth of penetration was approximately 0.0012 inch. However, as noted earlier, only a short time, on the order of 60 minutes, was required to cause the wetted fracture strength to drop to fairly low values. Microscopic examination of specimens exposed for this time showed but a slight roughening of the surface. The condition measured less than 0.0001 inch in depth.

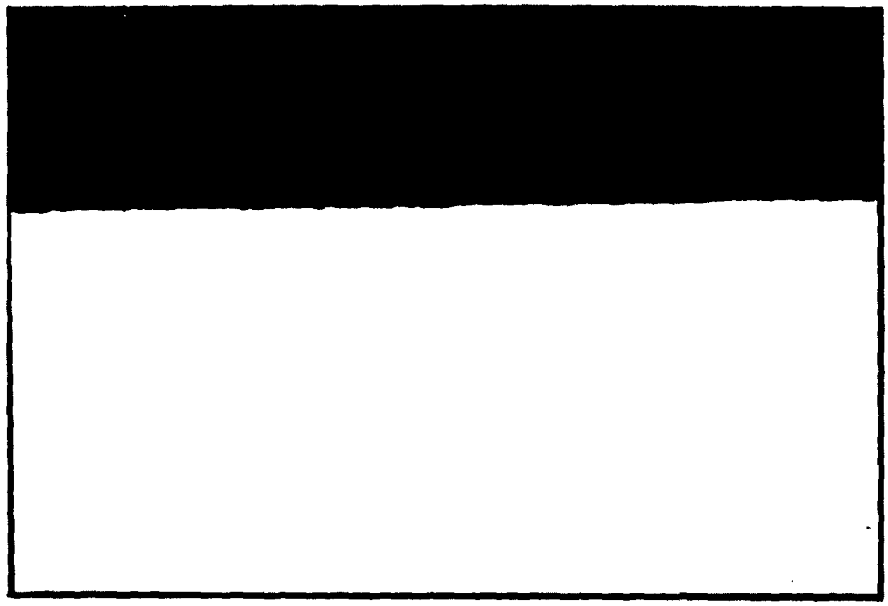
With longer times of exposure to amalgam, on the order of 48 hours, visible indications of intergranular cracking were noted along the surface of the aluminum. However, no such cracks were detected for the shorter times of exposure. This fact would seem to



Unetched

Keller's etch.

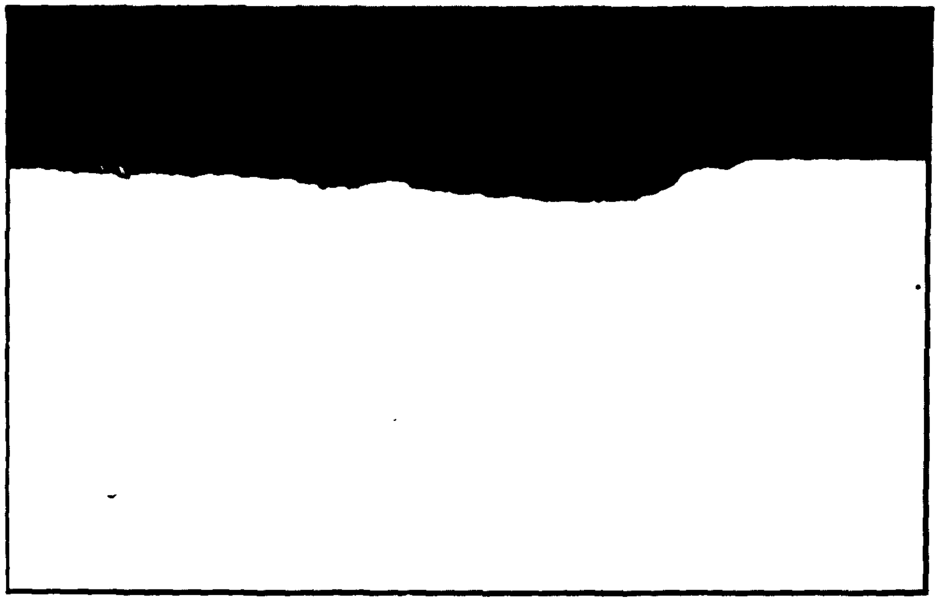
Figure 8. Photomicrographs depicting edge of 1/16 in. thick 2024 T-3 aluminum which had been wetted with 2% Zn - Hg amalgam and probe and then dewetted immediately. Transverse to rolling direction. Magnification 200X.



Unetched

Keller's etch.

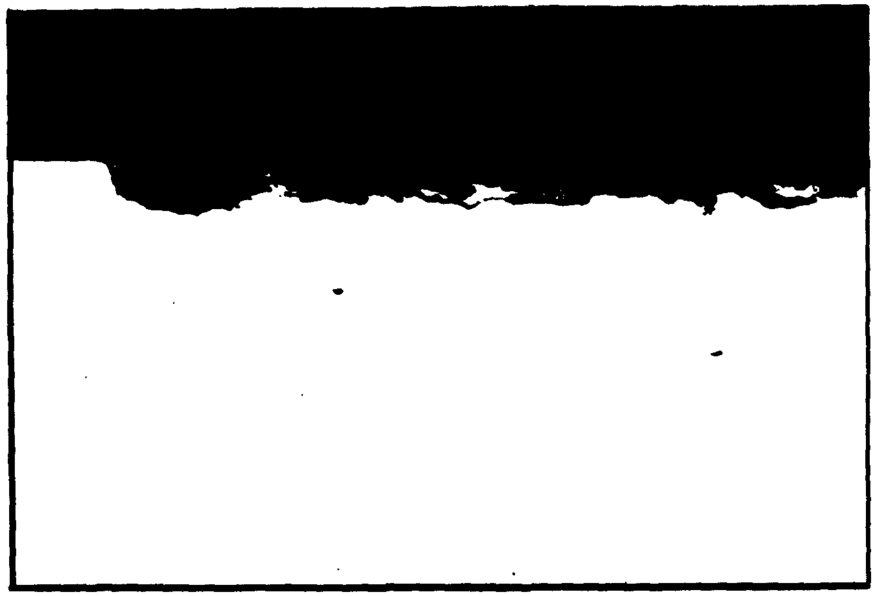
Figure 9. Photomicrographs depicting edge of 1/16 in. thick 2024 T-3 aluminum which had been wetted with 2% Zn - Hg amalgam and held two hours unstressed before dewetting. Transverse to rolling direction. Magnification 200X.



Unetched

Keller's etch.

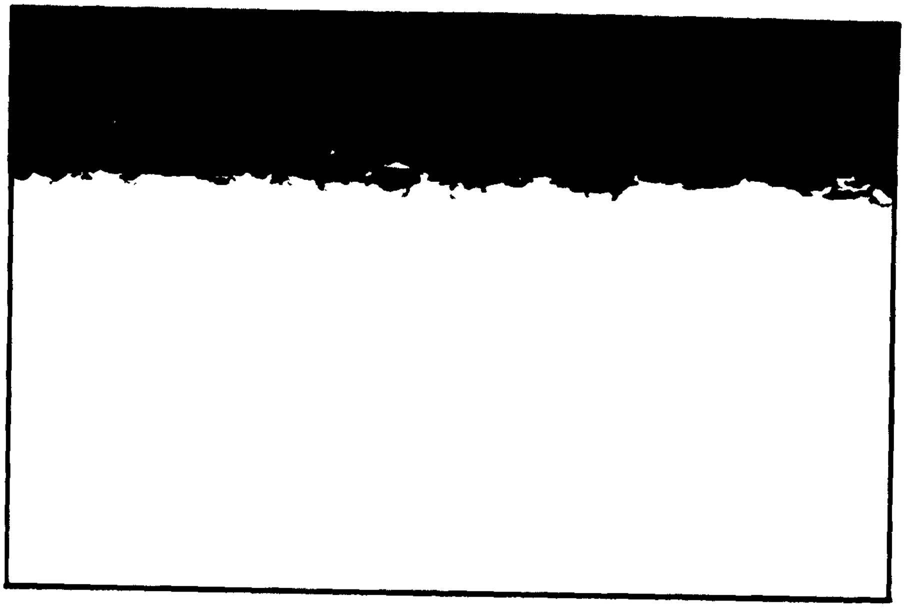
Figure 10. Photomicrographs depicting edge of 1/16 in. thick 2024 T-3 aluminum which had been wetted with 2% Zn - Hg amalgam and held eight hours unstressed before dewetting. Transverse to rolling direction. Magnification 200X.



Unetched

Keller's etch.

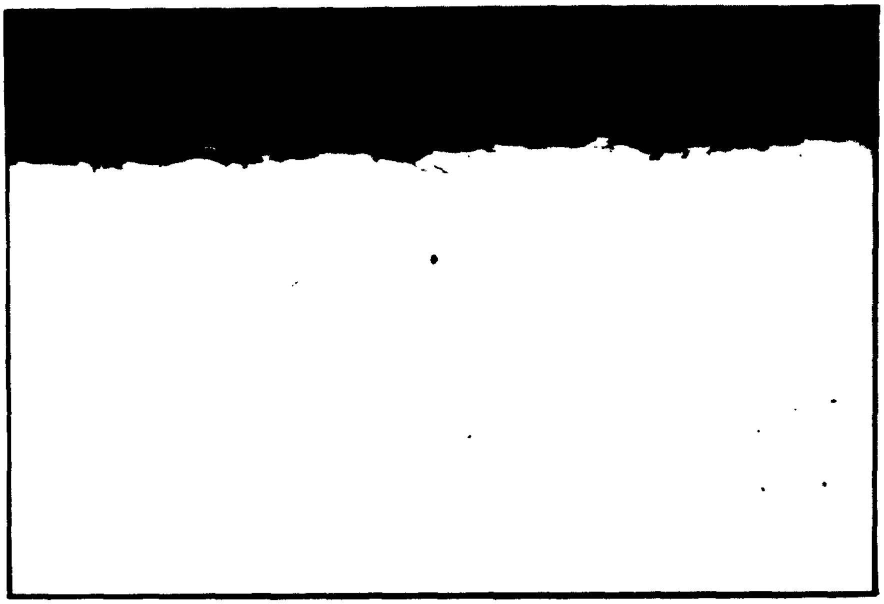
Figure 11. Photomicrographs depicting edge of 1/16 in. thick 2024 T-3 aluminum which had been wetted with 2% Zn - Hg amalgam and held 72 hours unstressed before dewetting. Transverse to rolling direction. Magnification 200X.



Unetched

Keller's Etch.

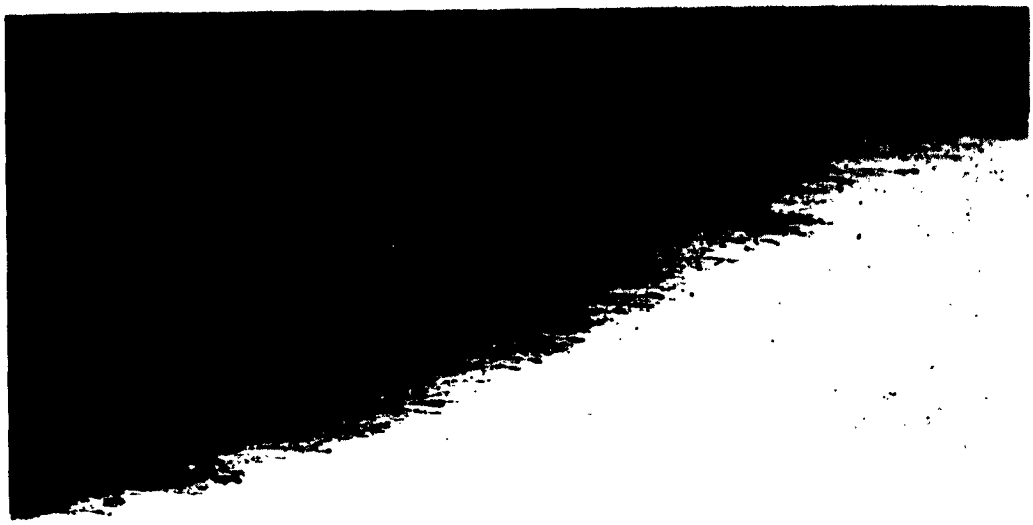
Figure 12. Photomicrographs depicting edge of 1/16 in. thick 2024 T-3 aluminum which had been wetted with 2% Zn - Hg amalgam and held 96 hours unstressed before dewetting. Transverse to rolling direction. Magnification 200X.



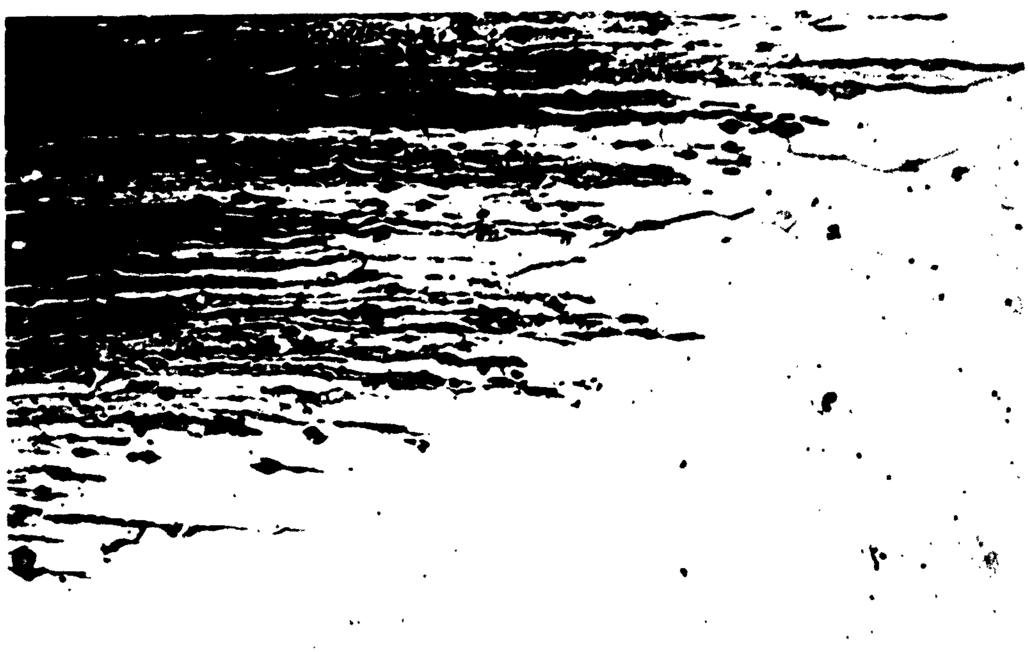
Unetched

Keller's etch.

Figure 13. Photomicrographs depicting edge of 1/16 in. thick 2024 T-3 aluminum which had been wetted with 2% Zn - Hg amalgam and held 7 days unstressed before dewetting. Transverse to rolling direction. Magnification 200X.



100X



500X

Figure 14. Photomicrographs depicting edge of 1/16 in. thick 2024 T-3 aluminum which had been wetted with 2% Zn - Hg amalgam and held 15 weeks unstressed before dewetting. Unetched.

indicate that increased embrittlement noted with increased time held unstressed is associated primarily with a surface effect and not the result of amalgam penetration into the unstressed aluminum.

Annealed and Strain Hardened Alloys

It was interesting to note that time dependence of wetted fracture strength is not caused solely by the aforementioned physical dissolution of the specimen surface with increased exposure to amalgam. Physical attack was observed on annealed unalloyed 1100 grade, on annealed 2024 and 7075 material and on strain hardening grade 5456, while none of these alloys exhibited any major time dependence of wetted fracture strength. Photomicrographs of these alloys showing extent of attack are shown in figures 15 and 16. Generally, the rate of dissolution for annealed and strain hardened material was comparable to that observed on precipitation hardened grades.

Clad Material

The time dependence of the wetted fracture strength for the Alclad 2024 T-3 material can best be understood from photomicrographs obtained on the pieces. For short time periods during which the wetted fracture strength was unchanged with time, the amalgam was separated from the age hardened aluminum by the unalloyed cladding. As noted earlier, unalloyed 1100 aluminum shows neither embrittling characteristics nor any decrease in wetted fracture strength with time. Eventually the cladding was in effect removed by dissolution and the amalgam made contact with the base metal so that the process could continue as with the bare age hardened material. The progressive dissolution of the cladding is depicted in figure 17.

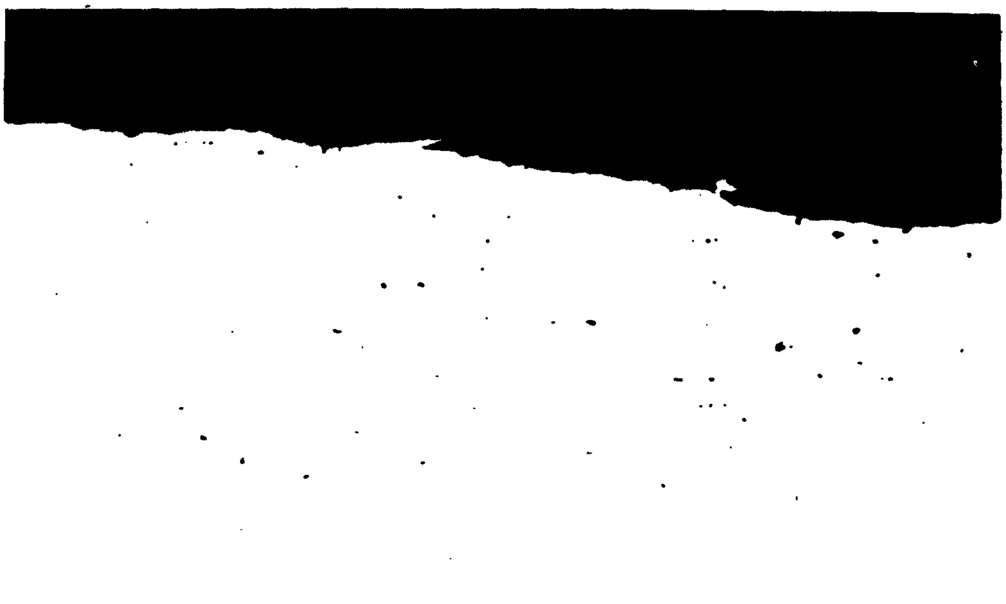
Mode of Fracture

With respect to an individual alloy and temper the mode of fracture did not appear dependent upon the stress level to which the specimens were subjected to produce failure. For 2024 T-3 material the appearance of the embrittled edges was noted and microstructural observations made of the method of crack propagation. No

Unetched

Keller's etch.

Figure 15. Photomicrographs showing edge of 2024-O aluminum sheet which had been wetted with 2% Zn - Hg amalgam and held five days unstressed before dewetting. Magnification 200X.



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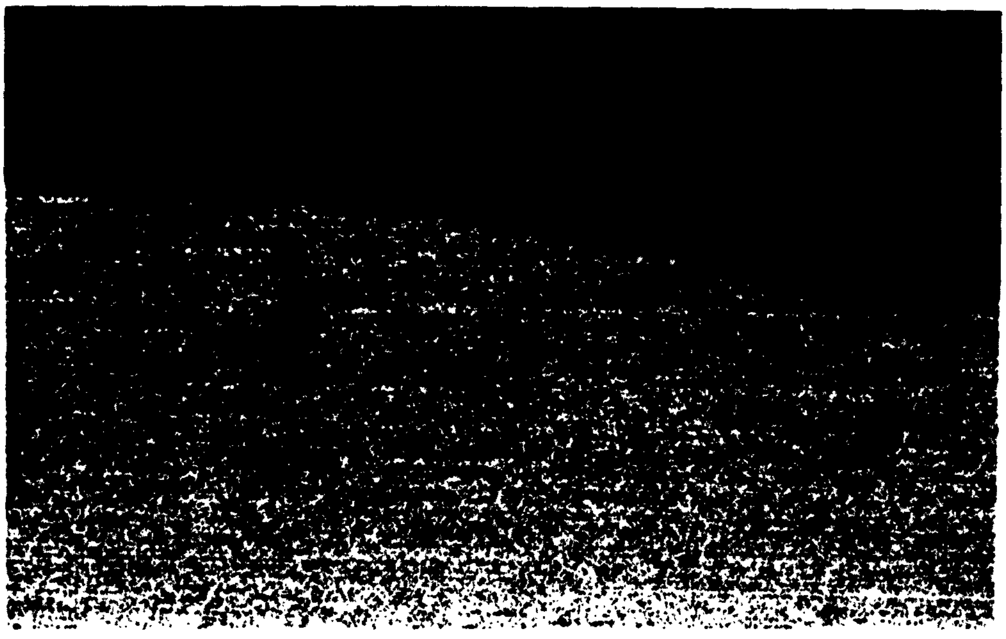
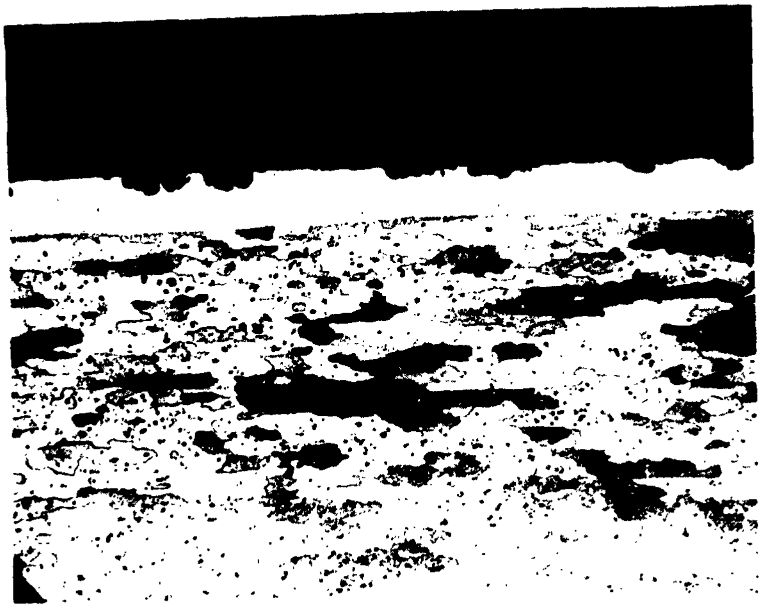


Keller's etch.

Figure 16. Photomicrographs showing edges of 0.090 in. thick 5456 H-24 aluminum which had been wetted with 2% Zn - Hg amalgam and ultrasonic probe and held 24 hours unstressed before dewetting. Magnification 200X.



15 minutes

1000 minutes

Figure 17. Photomicrographs showing edges of 1/16 in. thick 2024 T-3 Alclad aluminum which had been wetted with 2% Zn - Hg amalgam and ultrasonic probe and held unstressed for times indicated. Note progressive dissolution of cladding.
Magnification 100X, Keller's etch.

differences were seen between specimens which failed quickly upon application of high stress and those which failed at lower stress levels after prolonged holding times. Intergranular and transgranular cracking was evident in specimens from both conditions. Typical photomicrographs for each condition are shown in figure 18.

CONCLUSIONS

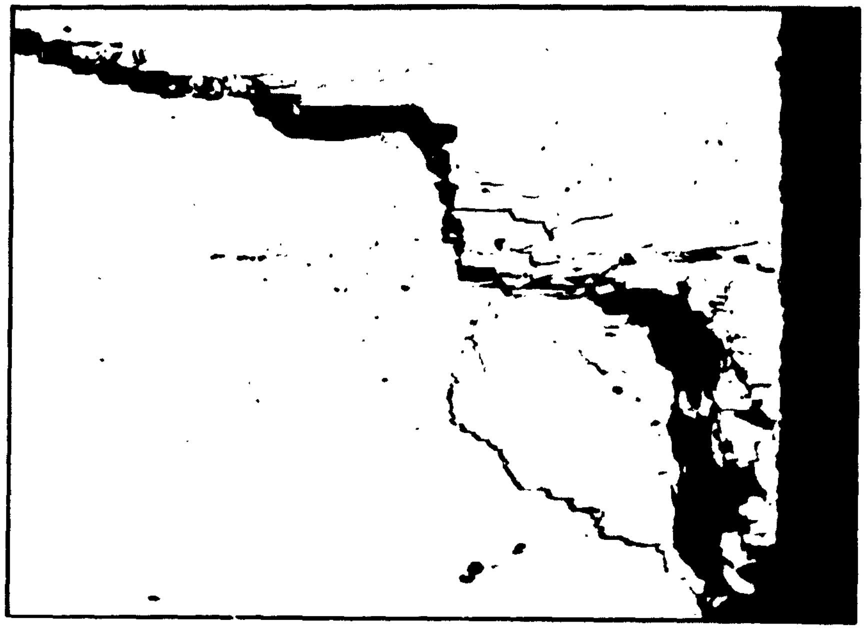
1. For the alloys and conditions studied, severity of embrittlement is dependent on the time of wetted contact between the aluminum and the amalgam before stressing.

2. This time dependence is most marked for alloys in the precipitation hardened condition in comparison to that for alloys in the annealed state or in the strain hardened temper.

3. Once wetting has been effected, there is a continuing physical dissolution attack of the alloys by the amalgam.

4. The rate of dissolution attack is approximately the same for all alloys and tempers.

5. For aluminum alloys in general no direct relationship has been established between the amount of dissolution and the degradation of fracture strength.



Wetted and tested immediately — W.F.S. = 46,000 psi

Held 24 hours, wetted, unstressed before test — W.F.S. = 8000 psi

Figure 18. Photomicrographs of failed edges of 1/16 in. thick 2024 T-3 aluminum sheet specimens upon embrittlement with 2% Zn - Hg amalgam. Note similarity of cracking for specimens which exhibited widely different wetted fracture strengths. Magnification 100X.

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2. W. Rostoker, "Fracture of Metals," Armour Research Foundation Report ARF 2183-8, September 1961.

3. B. J. Rogus, Report R-1677, Delayed Fracture of Aluminum Alloys by a Liquid Zinc Amalgam. Frankford Arsenal, April 1963.

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R-1678

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FRACTURE CHARACTERISTICS OF ALUMINUM ALLOYS WETTED WITH ZINC AMALGAM by B. J. Rogus.

Report R-1678, April 1963, 33 pp incl illustrations and tables, OMS 5010.11.80500.51-01
UNCLASSIFIED REPORT

Fracture strength determinations were made on sheet specimens of aluminum alloys 1100, 2024, 5456, 6061, and 7075 in different conditions of temper after wetting with a liquid 2% zinc amalgam. Tests at ambient temperature disclosed that the wetted fracture strength of the alloys decreased with increasing time of wetted contact with the amalgam before stressing. This time dependence was most pronounced for alloys in the precipitation hardened condition in comparison to that for the same alloys in the annealed state or others in strain hardened tempers.

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